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Rheological behavior of coal bio-oil slurries

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ABSTRACT

Coal bio-oil slurries (CBSs) were prepared by blending coal with bio-oil from the fast pyrolysis, and their apparent viscosities were measured by a rotary viscometer. The influences of coal rank, solid concentration, particle size distribution and temperature on the apparent viscosity and rheological properties of slurries were investigated. Additionally, the grey relational analysis was employed to determine the order of importance of factors affecting the apparent viscosity for different rank coals. Results show that, the CBS exhibits non-Newtonian fluid behavior and can be described by Herschel–Bulkley equation. The main factors for different rank coals affecting apparent viscosity of CBS are inherent moisture and carboxyl groups. The maximum solid concentration of CBS can reach 45–47 wt. % for Shenmu bituminous coal. Appropriate solid particle size distribution and preparation temperature can provide satisfied slurries with low viscosity.

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1. Introduction

Biomass is an important alternative renewable and carbon neutral energy resource compared to the fossil fuels [1–4]. However, direct application of biomass is restricted due to its higher transport cost and lower energy density [5]. So it is favorable to convert biomass into bio-oil by pyrolysis, and the energy density can be increased significantly. Bio-oil exhibits high oxygen content [6–8], high water content [7], strong acidity and instability [9], and all this makes it difficult for direct application. Thus, bio-oil needs to be upgraded for further utilization.

One of the effective applications of bio-oil is to make slurries with char or coal. The properties of bio-char based slurries have been studied by many researchers [10–12], and the suspensions could be used in combustion and gasification. As a replacement of coal water slurry (CWS), Wang [13] proposed the feasibility of making coal bio-oil slurry (CBS) using biomass fast pyrolysis oil and preliminarily tested its gasification performance. With coal added into bio-oil, the suspensions have much higher calorific value than bio-oil, and the bio-oil in turn contributes much more heat to CBS than CWS with the same coal concentration. Thus CBS could also be used in combustion process and syngas production by gasification. Moreover the bio-oil derived from biomass in CBS does not increase CO_2 emission. Therefore, it could be considered as a partial green fuel.

In combustion and gasification process the slurry needs to keep high coal concentration for high calorific heat or syngas yield. It requires optimal solid loadings with low apparent viscosity simultaneously. Therefore, rheological behavior is an important parameter for pumping and atomizing of CBS slurry. The purpose of the present work is to investigate the effect of coal ranks (from lignite to anthracite), coal concentration and particle size distribution on the apparent viscosity and viscosity–temperature properties of CBS.

2. Experimental section

2.1. Materials

Bio-oil sample used in the study was from Shandong Yineng Bioenergy Co., Ltd, China. It was produced in a fluidized bed by pyrolysing rice husk at around 500 °C and its properties were summarized in Table 1. Elemental composition of bio-oil was measured by an elemental analyzer instrument (vario EL cube). The water content and pH value of bio-oil were determined by Metrohm Karl Fischer Titrino and a pH meter, respectively. The higher heating value (HHV) of bio-oil was determined by a calorimeter (XRY-1B, Shanghai Changji Instrument), and the lower heating value (LHV) of bio-oil was calculated by Eq. (1) [14,15].

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Nomenclature

CBS	coal bio-oil slurry
CWS	coal water slurry

$$\mathrm{LHV[J/g]} = \mathrm{HHV[J/g]} - 218.13 \times H\%\mathrm{[wt\%]} \tag{1}$$

Ten coal samples used in this work are from different areas, and their ranks are from lignite to anthracite. The proximate analysis and ultimate analysis of these coals are given in Table 2. Coals are ball milled to give the similar particle size distributions as shown in Fig. 1.

When investigating the influence of particle size distribution, coal from Shenmu (SM) was crushed and then ball milled for different time to obtain samples with different particle size distributions noted as S-PSD1, S-PSD2, S-PSD3 in Fig. 2(a). Samples with different proportions of S-PSD3 mixed with S-PSD1 are shown in Fig. 2(b). Coal samples were then dried at 105 °C for 4 h. Both higher heating value (HHV) and lower heating value (LHV) of SM coal (dry basis) were analyzed by a calorimeter (XRY-1B, Shanghai Changji Instrument). The HHV and LHV of SM coal are 22,724 and 21,815 J/g respectively.

2.2. Experiment and methods

Coal bio-oil slurries with different solid concentrations were prepared by adding coal particles into 100 g of bio-oil, and then they were stirred with a high speed agitator for 30 min until a homogeneous slurry formed. No emulsions were added in CBS.

Total acidic functional groups and carboxyl groups of coal samples were determined by $Ba(OH)_2$ and $Ca(CH_3COO)_2$, respectively, and the content of phenolic hydroxyl groups were calculated by subtraction [16,17].

The low-temperature nitrogen adsorption analysis was carried out in Quantachrome automatic physical and chemical adsorption instrument (AUTOSORB-1-C-TCD). All samples were measured at 77 K using nitrogen as adsorbate to obtain BET surface area and total pore volume.

The apparent viscosity of coal bio-oil slurries was measured by a rotary viscometer (NXS-11B, Chengdu Instrument Factory, China). A water bath was used to keep constant temperature of slurry samples. The details of apparent viscosity of CBS measurement were corresponded with the method of coal water slurry [18]. The experimental error of apparent viscosity was estimated based on the law of error propagation, which involves how random errors in the experimental measurements are propagated into errors in the quantity calculated according to the measurements [19]. The apparent viscosity was a function of shear stress as defined in Eq. (2), and the shear stress can be calculated from Eq. (3).

$$\eta = \frac{\tau}{\gamma} \times 1000 \tag{2}$$

where η is apparent viscosity, mPa s; τ is shear stress, Pa; γ is shear rate, s^{-1}.

$$\tau = Z \times \alpha \tag{3}$$

where Z is a constant coefficient (0.5675) which does not change with shear rate, Pa; α is the reading in dial of the viscometer.

At certain shear rate, the apparent viscosity (η) is only determined by α. Thus the limit of error of η can be calculated based on equation (4).

$$\sigma_\eta^2 = \frac{Z}{\gamma}\delta_\alpha^2 \tag{4}$$

where σ_η^2 and δ_α^2 are the limits of error of η and α respectively, and δ_α is 0.1 for the viscometer. σ_η^2 at different shear rates are listed in Table 3. As shown in Table 3, the limits of error of apparent viscosity at all shear rates are in the range of 0.1538–1.7857, which is rather

Table 1
The physical properties and elemental composition of bio-oil sample.

Physical properties	
Water content (wt.%)	43.77
pH	2.71
Viscosity at 25 °C (mPa·s)	9.23
HHV[a] (J/g)	12,481
LHV[b] (J/g)	10,692
Elemental composition (wt%)	
C	30.16
H	8.20
O^{diff}	60.64
N	0.96
S	0.04

[a] HHV, higher heating value.
[b] LHV, lower heating value.

Table 2
Proximate and ultimate analysis of coal samples.

Coal sample	$M_{inh,ar}$[a]	Proximate analysis (wt%)			Ultimate analysis (wt%)				
		V_d[b]	A_d[c]	FC_d[d]	C_d	H_d	N_d	O_d[e]	S_d
GC	3.94	31.95	8.64	59.41	74.98	4.10	1.05	10.87	0.35
YL	4.13	28.79	47.06	24.15	36.45	3.31	0.93	11.90	0.35
XL	9.87	31.79	26.39	41.82	54.96	3.86	0.82	12.99	0.98
FG	4.89	36.11	4.49	59.40	76.43	4.30	1.08	13.58	0.12
TX	0.85	11.19	5.29	83.52	87.01	3.20	0.70	3.55	0.26
BR	8.48	44.00	11.96	44.04	62.60	4.28	0.84	20.05	0.26
HN	1.57	10.36	24.14	65.50	67.03	2.37	0.61	3.69	2.16
TY	1.78	25.49	31.05	43.46	53.12	3.13	0.94	11.30	0.47
TS	1.22	23.58	30.40	46.02	57.76	3.20	0.94	6.81	0.88
SM	4.29	28.35	25.29	46.36	59.88	3.59	0.71	10.12	0.40

[a] Inherent moisture content in as received basis.
[b] Volatile content in dry basis.
[c] Ash content in dry basis.
[d] Fixed carbon content in dry basis.
[e] Oxygen content is calculated by difference. We assume that it does not include oxygen contained in ash.

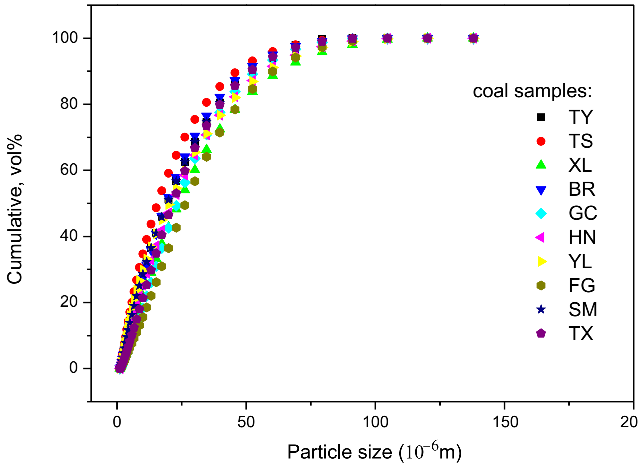

Fig. 1. Particle size distributions (PSD) of ten coal samples.

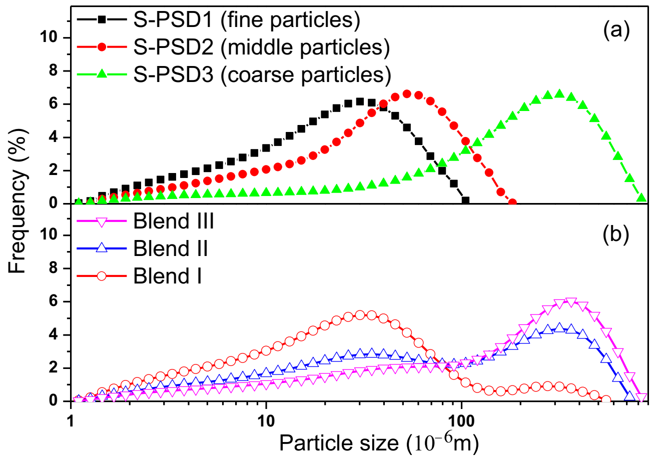

Fig. 2. Particle size distributions of Shenmu (SM) bituminous coal. (a) Three distributions of ball milled coal samples. (b) Coal samples made by different proportions of S-PSD1 (fine particles) and S-PSD3 (coarse particles). The mass ratio of S-PSD1 (fine particles) and S-PSD3 (coarse particles) are: Blend I, 70:30; Blend II, 50:50; Blend III, 30:70).

small compared to the apparent viscosity. Therefore it will not affect the reliability of the experimental results.

3. Results and discussion

3.1. Rheological behavior of coal bio-oil slurry

In the study, the solid loadings of CBS were prepared from 30 wt. % to 47 wt. %, and the slurries exhibit typical non-Newtonian fluid behavior. In general, non-Newtonian fluids could be described by different equations:

Power Law:

$$\tau = k\dot{\gamma}^{m} \quad \text{or} \quad \eta = k\dot{\gamma}^{m-1} \tag{5}$$

Bingham plastic:

$$\tau = \tau_{\mathrm{B}} + \eta_{\mathrm{B}}\dot{\gamma} \quad \text{or} \quad \eta = \frac{\tau_{\mathrm{B}}}{\dot{\gamma}} + \eta_{\mathrm{B}} \tag{6}$$

Herschel–Bulkley:

$$\tau = \tau_{\mathrm{H}} + k'\dot{\gamma}^{n} \quad \text{or} \quad \eta = \frac{\tau_{\mathrm{H}}}{\dot{\gamma}} + k'\dot{\gamma}^{n-1} \tag{7}$$

where τ means the shear stress, Pa; η is the apparent viscosity, Pa·s; $\dot{\gamma}$ is the shear rate, s^{-1}; m, n are the Power law model index and the Herschel–Bulkley model index respectively; k, k' are consistency parameters; τ_{B}, τ_{H} are the yield stress.

To determine the flow behavior of slurries, the regression for all samples of shear stress and shear rate was analyzed, and Table 4 gives the results of CBS (35 wt% coal concentration) made of four different rank coals. Results indicated that it has a highly fitting accuracy of Herschel–Bulkley model with the R-square closest to 1. So Herschel–Bulkley was used to describe the rheological behavior of CBS.

Table 4
The regression results of models.

Coal sample used in slurries	R-square of models		
	Power law	Bingham plastic	Herschel–Bulkley
BR	0.9890	0.9871	0.9996
SM	0.9925	0.9926	0.9977
TY	0.9707	0.9996	0.9996
HN	0.9947	0.9742	0.9996

3.2. Effect of coal properties on the apparent viscosity and rheological behaviors

3.2.1. Coal ranks

Coal rank is a significant factor influencing the apparent viscosity of slurries [20]. Researches have proved that the apparent viscosity of slurry could be affected by O/C [21,22], inherent moisture ($M_{\mathrm{inh,ar}}$) [21,23], ash content (A_{d}) [21], oxygen-containing functional groups [21] (total acidic groups, carboxyl groups and phenolic hydroxyl groups), and pore structure [23,25] (BET surface area, total pore volume) of coal, and all this are changing with coal ranks. In this work, grey relational analysis is employed to determine the main factors affecting the apparent viscosity of CBS. Grey relational analysis is a method based on grey system to determine the main factors. If the trends of two factors are similar, it is believed that their correlation is greater than others [26]. The similarity is expressed by the grey relational grade.

The data processing is similar to others [27,28]. The first step of grey relational analysis is to determine the reference sequence Y_0, $Y_0 = [Y_0(1), Y_0(2),\ldots, Y_0(k)]$, which refers to the apparent viscosity of CBS (35 wt% coal concentration) at 100 s^{-1}. The coal properties are then set as compared sequences $Y_i = [Y_i(1), Y_i(2), \ldots, Y_i(k)]$. All sequences are shown in Table 5.

For all sequences, since their units or initialization are different, they need to be treated by as Eq. (8).

$$X_i(k) = \frac{Y_i(k)}{\frac{1}{n}\sum\limits_{k=1}^{n} Y_i(k)} \tag{8}$$

where k is the number of coal samples; n is the number of compared factors.

For each compared sequence, $|X_0(k) - X_i(k)|$ can be calculated. Also $\min\limits_{i}\min\limits_{k}|X_0(k)-X_i(k)|$ and $\max\limits_{i}\max\limits_{k}|X_0(k)-X_i(k)|$ are obtained. The grey relation coefficient can be expressed as

$$\xi_i(k) = \frac{\min\limits_{i}\min\limits_{k}|X_0(k)-X_i(k)| + \rho\max\limits_{i}\max\limits_{k}|X_0(k)-X_i(k)|}{|X_0(k)-X_i(k)| + \rho\max\limits_{i}\max\limits_{k}|X_0(k)-X_i(k)|} \tag{9}$$

where ρ is the distinguishing coefficient set between zero to one, and it was set to 0.5 in this study.

The grey relational grade is defined as

$$\gamma_i = \frac{1}{n}\sum_{k=1}^{n}\xi_i(k) \tag{10}$$

The grey relational grade calculated is given in Table 6.

Table 3
The limits of error (σ_η^2) of apparent viscosity at different shear rates.

γ	3.178	4.313	5.675	7.378	10.22	15.89	21.57	28.38	36.89
σ_η^2	1.7857	1.3157	1.0000	0.7592	0.5553	0.3571	0.2631	0.2000	0.1538

Table 5
The reference sequence and compared sequences.

Coal sample	Apparent viscosity at 100 s^{-1} (mPa s)	O/C[e] (%)	$M_{inh, ar}$[f] (%)	A_d[g] (%)	BET surface area (m^2/g)	Total pore volume (mL/g)	Total acidic functional groups (mmol/g)	Carboxyl groups (mmol/g)	Phenolic hydroxyl groups (mmol/g)
XL[a]	4134.0240	23.6422	9.8700	26.3900	8.8600	0.0361	2.9000	0.7505	2.1495
BR[a]	1809.3861	32.0300	8.4800	11.9600	7.4480	0.0302	4.6982	1.2294	3.4688
YL[b]	65.7261	32.6560	4.1300	47.0600	19.9000	0.0619	1.3491	0.2117	1.1374
FG[c]	456.2777	17.7717	4.8900	4.4900	9.4230	0.0232	1.9037	0.2992	1.6045
GC[c]	489.6425	14.4940	3.9400	8.6400	20.7000	0.0344	1.8634	0.2951	1.5683
SM[c]	169.8036	16.9080	4.2900	25.2900	11.8000	0.0283	1.5096	0.2876	1.2220
TS[c]	110.3892	11.7946	1.2200	30.4000	5.5200	0.0238	0.2221	0.0754	0.1467
HN[d]	72.7348	5.5017	1.5700	24.1400	187.0000	0.2170	0.8731	0.1565	0.7166
TX[d]	193.2170	4.0802	0.8500	5.2900	6.5000	0.0139	0.0649	0.0509	0.0140
TY[b]	183.8803	21.2766	1.7800	31.0500	9.7000	0.0281	0.8565	0.1565	0.7000

[a] Lignite.
[b] Long flame coal.
[c] Bituminous coal.
[d] Anthracite.
[e] Mass ratio of oxygen to carbon.
[f] Inherent moisture content in as received basis.
[g] Ash content in dry basis.

It can be seen in Table 5 that CBS made by lignite is of higher viscosity than other rank coals. As listed in Table 6, the order of importance of the coal characters to the apparent viscosity of CBS in sequence can be listed as: Carboxyl groups > $M_{inh, ar}$ > Total acidic groups > Phenolic hydroxyl groups > O/C > BET surface area > Total pore volume > A_d. The grey relational grade of $M_{inh, ar}$ and carboxyl groups in coal are greater than others. This indicates that the apparent viscosity of CBS was strongly affected by $M_{inh, ar}$ and carboxyl groups in coal. Moisture has been proved by many researchers to be an important index of the hydrophilicity of a coal [22–24]. Another aspect which can also be used to represent the hydrophilicity is the oxygen-containing functional groups. In coal water slurry, the more hydrophilicity of a coal, the larger amount of water can be absorbed by the coal, and thus less water is used as a fluidizing medium [23]. In coal bio-oil slurry, the suspension medium is changed to bio-oil. Bio-oil is composed of water and some water-soluble organic compounds with a small amount of water-insoluble materials [9], and consequently bio-oil is of high polarity. The polar component, i.e. water and some other polar molecules, tends to be absorbed on the surface of hydrophilic coal particles. So if a coal is hydrophilic, it is likely to hold bio-oil, and less fluid is left to flow. Therefore the apparent viscosity increases. Typically, high rank coals is relatively low in moisture and oxygen-containing functional groups, hence CBS made by high rank coals exhibits low apparent viscosity.

3.2.2. *Coal concentration*

The apparent viscosity of CBS as a function of solid concentration at room temperature (25.0 ± 0.1 °C) was showed in Fig. 3. The results indicate that the coal concentration has an obvious influence on the apparent viscosity of CBS. The apparent viscosity decreases with increasing shear rate, and the fluid exhibits an obvious shear thinning behavior due to flocculated structures [13,29] formed in the slurries. The nonlinear relation between apparent viscosity and coal concentration at shear rate of 100 s^{-1} was also shown in Fig. 3. The increment of apparent viscosity from 40 wt% to 45 wt% is about two times higher than that from 35 wt% to 40 wt%, and this means a little increase in solid concentration would cause a dramatic rise in apparent viscosity when the solid loading is above 40 wt%. In addition, the apparent viscosity of the CBS with 45 wt% is 1000 mPa s at 100 s^{-1}, and it is approximate 1300 mPa s of the slurry with 47 wt%. In terms of the definition of the maximum solid concentration [18], the range of CBS for SM coal is about 45 wt% to 47 wt%.

The low heating value (LHV) is a key evaluation index for the fuels, and the coal concentration is a main factor influencing the LHV of slurries. Taking the CBS in concentration of 45 wt% as an example, the LHV is 15.70 kJ/g, equivalent to the solid concentration of CWS of 71.96 wt% with the same LHV, while the CWS in this concentration can hardly be a flowing slurry. So it is worthy to note

Table 6
Grey relation grade and its order of factors in different rank coals.

Factors	Grey relational grade	Order
$M_{inh, ar}$[a]	0.8554	2
A_d[b]	0.7426	8
BET surface area	0.7953	6
Total pore volume	0.7871	7
Total acidic groups	0.8499	3
Carboxyl groups	0.8689	1
Phenolic hydroxyl groups	0.8452	4
O/C[c]	0.8163	5

[a] Inherent moisture content in as received basis.
[b] Ash content in dry basis.
[c] Mass ratio of oxygen to carbon.

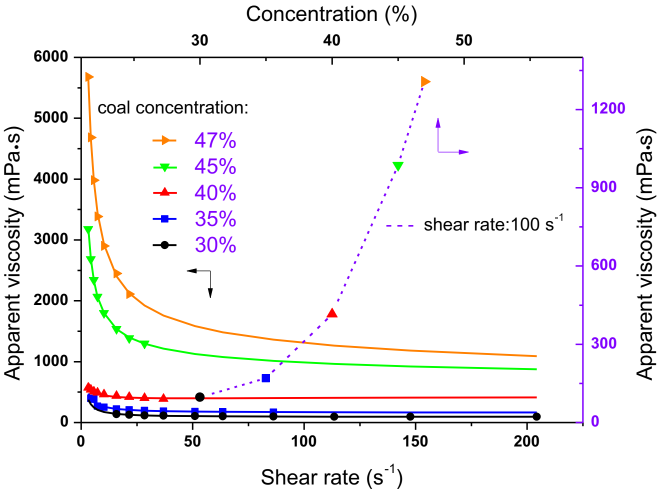

Fig. 3. The apparent viscosity changes of coal bio-oil slurries with coal concentrations at 25 °C (Sample: S-PSD2 (middle particles). The solid lines represent a model plot of Herschel–Bulkley fluid.).

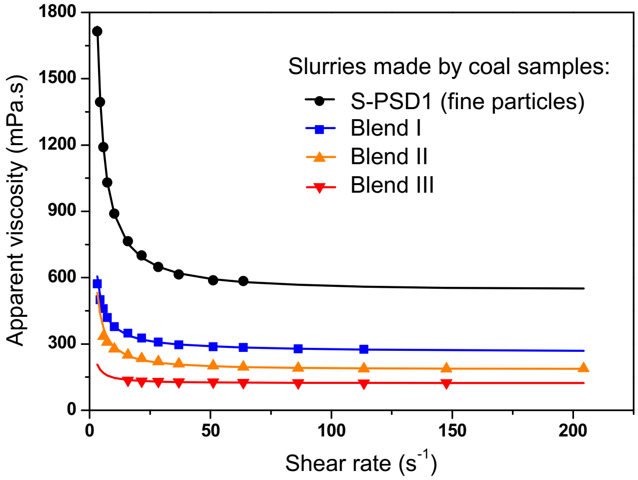

Fig. 4. Effect of coal particle size distribution on the apparent viscosity of slurries at 25 °C (The slurries are made by coal samples with different ratios of fine particles to coarse particles: S-PSD1, 100:0; Blend I, 70:30; Blend II, 50:50; Blend III, 30:70. Coal concentration: 40 wt. %. The solid lines represent a model plot of Herschel–Bulkley fluid.).

that under the condition of same LHV, the flowability of CBS was of much lower apparent viscosity than that of CWS. Furthermore, the LHV of CBS is significantly increased compared with bio-oil. So the CBS is more promising to be applied in the combustion or gasification process.

3.2.3. *Coal particle size distribution*

Fig. 4 shows the effect of coal particle size distribution on the apparent viscosities of CBS with solid concentration of 40 wt. % at 25 °C. Solid coal particles in slurries were prepared by adding coarse particles (S-PSD3) into fine particles (S-PSD1), and the data of particle size distributions was described in Fig. 2(b), and respective mean grain size was listed in Table 7. It can be seen from Table 7 that the apparent viscosity decreases as the mean grain size increasing, and the slurry sample Blend III demonstrated the lowest apparent viscosity and an optimal particle size distribution. This could be explained that void fraction in solid coal particles differs with different particle size distributions. The correlation of void fraction (ε) and pack efficiency (λ) is $\lambda + \varepsilon = 1.0$ [18], in which the pack efficiency can be reflected by the pack volume. The pack volume was measured as the reference [25] described and the tested results were listed in Table 7. It is noted that under the constant solid loading, the pack volume decreases with the blending ratio of the coarse particles increasing. The coal particles in the slurry sample Blend III with the lowest void fraction have the highest pack efficiency, and this means that the least amount of liquid bio-oil is required to fill the inter-particle voids. Increasing of volume of flow phase in the slurry, thereby reduce the viscosity of CBS.

3.3. *Viscosity–temperature properties of the CBS*

Temperature must be considered in the transportation process of slurries. The effects of temperature between 25 and 70 °C on viscosities of bio-oil and coal bio-oil slurry were investigated, and the results were shown in Fig. 5. The viscosity of bio-oil keeps constant at a certain temperature with the shear rate changing, thus bio-oil exhibits an essential Newtonian behavior as other pyrolysis liquids [15,30]. When the temperature increases from 25 to 70 °C, the apparent viscosity of CBS decreases dramatically from 387.58 mPa·s to 87.67 mPa·s at 100 s^{-1}. A distinct increase of the apparent viscosity occurred at temperature in the range of 30–40 °C. For CBS, the apparent viscosity also significantly decreases in range of 30–40 °C. The variation of viscosity of bio-oil and CBS is quite similar with temperature changing, which means that the effect of temperature on viscosity of CBS strongly depends on the properties of bio-oil.

Table 7
The pack volume for Shenmu (SM) bituminous coal of the same weight with different fractions of coarse particles.

Slurry sample	S-PSD1	Blend I	Blend II	Blend III
Pack volume (mL)	8.018	7.135	6.596	6.378
Mean particle size (μm)	9.93	13.52	17.34	25.22

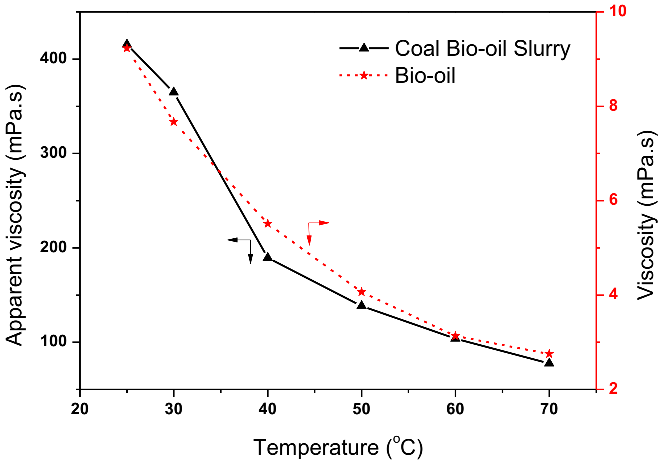

Fig. 5. The viscosity–temperature behaviors of bio-oil and coal bio-oil slurry (Coal concentration of coal bio-oil slurry: 40 wt. %, Sample: S-PSD2 (middle particles), shear rate: 100 s^{-1}).

4. Conclusions

The rheological behaviors of CBSs with solid concentrations of 30–47 wt. % were investigated in the present study, and a typical non-Newtonian fluid behavior was identified which can be described by Herschel–Bulkley equation. Their rheological properties can be described by Herschel–Bulkley equation. The apparent viscosities of CBS vary with coal ranks, coal concentration, solid particle size distribution and temperature. CBS made by high rank coals exhibits lower apparent viscosity. Based on grey relational analysis, inherent moisture and carboxyl groups in coal are determined as main factors affecting the apparent viscosity of CBS for different rank coals. The maximum solid concentration can reach 45–47 wt. % for SM coal, and the LHV is equal to that of CWS with 71.96 wt. % coal concentration. Appropriate solid particle size distribution and higher temperature are beneficial to reduce the apparent viscosity of CBS.

Acknowledgments

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